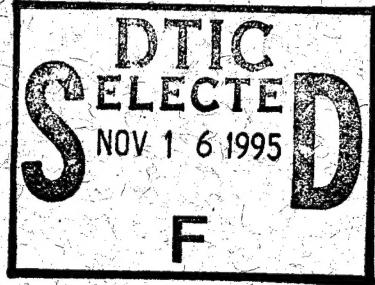


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**Spectroscopic Comparison
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Edward R. Long, Jr.,
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Hampton, Virginia*

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Summary

This report compares the differences in the radiation durabilities of two polyimide materials, Du Pont Kapton and General Electric Ultem. It provides an explanation of the basic mechanisms which occur during exposure to electron radiation from analyses of infrared (IR) and electron paramagnetic resonance (EPR) spectroscopic data for each material.

The molecular model for Kapton was, in part, established from earlier modeling for Ultem (pp. 1293-1298 of *IEEE Transactions on Nuclear Science*, December 1984). In this respect, the report also demonstrates techniques for understanding the durability of one complex polymer based on the understanding of a different and equally complex polymer.

The spectroscopic data showed that the primary radiation-generated change in the tensile properties of Ultem (a large reduction in tensile elongation) was due to crosslinking, which followed the capture by phenyl radicals of hydrogen atoms removed from gem-dimethyl groups. In contrast, the tensile properties of Kapton remained unchanged because radical-radical recombination, a self-mending process, took place.

Introduction

The high specific strengths and stiffnesses and the low thermal expansion coefficients of graphite-fiber-reinforced polymer-matrix composites are desired qualities for space structural applications. Polymer films are useful in space as low density insulations and solar-cell substrates. Long-term stability in space radiation environments is an important consideration for both of these applications. Polyimides are among the polymers being seriously considered for future space applications because they constitute a generic polymer class that maintains desirable mechanical properties at temperatures above 300°C. Reference 1 provides a general review of the radiation literature available, as well as references to research pertaining to polyimides.

A polyimide currently used in space applications is the polypyromellitimide commercially available under the name Kapton¹. Laboratory studies have shown (refs. 2 and 3) that the tensile properties of Kapton film are relatively unaffected by ionizing radiation. However, this polymer is not available in the forms of castings or composites because of processing problems associated with the removal of solvents from thick cross sections. Therefore, structural components cannot be made from this material.

¹ Kapton is a registered trademark of the E. I. du Pont de Nemours & Company, Inc.

A new class of polyimides, known as polyetherimides, can be used for composites and castings. One polyetherimide is commercially available under the name Ultem². Polyetherimides are thermoplastics, yet they retain much of the high-temperature performance of the thermosets. However, a recent study (ref. 4) has shown that, at least in the neat resin form, these new polymers are less durable to electron radiation than Kapton.

In this report, data are presented and compared for the effects of electron radiation doses expected for 30-year missions in geosynchronous orbit on the molecular structures and tensile properties of Kapton and Ultem. A model presented in an earlier report (ref. 4) that related radiation-generated changes in molecular structure and tensile properties for Ultem is used in this report to provide the basis for a model that relates the changes in molecular structure and tensile properties for Kapton. In this respect, the report demonstrates techniques for understanding the durability of one complex polymer based on the understanding of a different and equally complex polymer.

Materials and Specimens

Kapton is a product of the E. I. du Pont de Nemours & Company, Inc. The Kapton film for this study was type H-film and was used in two thicknesses, 0.003 inch and 0.00025 inch. Ultem is a product of the General Electric Company. The Ultem film for this study was type 1000 and was also used in two thicknesses, 0.003 inch and 0.0003 inch. The thicker films were used for the tensile specimens and the electron paramagnetic resonance (EPR) specimens. The thinner films were used for the transmission infrared (IR) specimens.

The tensile specimens were 0.200 ± 0.001 inch wide and 6.0 ± 0.1 inches long. The EPR specimens were 0.100 ± 0.001 inch wide and 0.600 ± 0.003 inch long. Both types of specimens were fabricated from larger pieces of the film by using dual razor blade cutters that are discussed in reference 1. The only size requirement for the IR specimens was that they be sufficiently large to cover the 0.25- by 1.0-inch opening in the IR specimen holder.

Experimental Arrangement

The experimental procedure for this study consisted of simultaneous exposures of mechanical and spectroscopic specimens of both Kapton and Ultem to 100-keV electron radiation for the selected total

² Ultem is a registered trademark of the General Electric Company.

doses of 1.6, 4.0, and 6.0 gigarad at a dose rate of 1 gigarad per hour. The specimen temperature during exposure for this dose rate was between 35°C and 38°C (ref. 1). This is the range of total dose that has been estimated for 30 years in geosynchronous Earth orbit (GEO). The specimens were placed in liquid nitrogen within 3 minutes of the completion of an exposure to retain the molecular structural components present at that time. For the EPR tests, this meant that all the unpaired electrons (radicals), except those with half lives of seconds or fractions of seconds, were trapped and their spectra could be recorded. The testing began immediately thereafter.

The tensile specimens were brought to room temperature, one at a time, and tested by using a model 1130 table-top Instron universal mechanical tester. The specimen grips were rubber surfaced, and their separation during each test was monitored with a direct current displacement transducer (DCDT). The outputs of DCDT and the load cell were recorded on an *X-Y* recorder. The crosshead rate was 0.2 inch per minute. A minimum of six specimens per exposure were tested.

The EPR specimens were placed in quartz glass tubes. The tubes were evacuated to 10^{-8} torr and sealed off by a propane torch, while the portion of the tube containing the specimen was kept in liquid nitrogen. These ampoules were kept in the liquid nitrogen until the first EPR scan, during which they were maintained in a thermal environment of -188°C . The EPR spectrometer, which detects unpaired electrons (radicals), was a Varian E-Line Century Series Model E-109E. The EPR spectrometer system included a variable temperature accessory for controlling the temperature during the scans, a precision microwave frequency counter, and a gaussmeter. Following the first scan, the ampoules were brought to room temperature for a designated period of time and then stored back in the liquid nitrogen. Then, another cycle of EPR scanning, followed by a specified time interval at room temperature, was conducted. In this manner, the postirradiation radical decay at room temperature in vacuum was measured.

The IR specimens were each mounted with Kapton polyimide tape on a thin rectangular brass frame. The irradiated specimens were placed in a Perkin-Elmer model 599B IR spectrometer and scanned from 4000 to 200 cm^{-1} at room temperature. The spectrometer was controlled by a Perkin-Elmer Model 3600 data station. No attempt was made to monitor the IR signal as a function of the time after exposure.

More complete descriptions of the radiation facility, the testing equipment, the specimens, and the manner in which they were prepared and handled

are given in reference 1. The methods used for monitoring the dose rate and the specimen temperature and other experimental details are also described in reference 1.

Results and Discussion

The major effect of the radiation on the tensile properties of the Ultem was a large reduction in the total elongation to failure (refs. 1 and 4), as presented in figure 1. The 1σ (one standard deviation) variations for these data were from ± 0.7 to ± 1.2 percent. The effects on the ultimate stress and the modulus were not significant, as shown in figures 2 and 3, respectively. The 1σ variations for these data were from ± 0.5 to ± 0.8 ksi for the ultimate stress and from ± 12 to ± 40 ksi for the modulus. These errors are too small to show clearly on the data points in the figures. In contrast to the Ultem, the Kapton, whose data are also shown in figures 1 through 3, showed no changes in its tensile properties for the range of total doses from 1.6 to 6.0 gigarad (ref. 5). The 1σ variations for the Kapton data were less than those for Ultem.

Polymeric materials are affected by radiation by two different phenomena that occur at the molecular level. One of these phenomena is crosslinking, and the other is chain scissioning. Therefore, for the Kapton, either the two phenomena occur in equal amounts in such a manner that the mechanical properties remain unchanged, or a mending process occurs such that there is little or no change in the molecular structure. Other characterization methods, such as spectroscopic analyses, are required for determining which basic mechanisms take place.

The molecular structures of the repeat units for Ultem and Kapton are given in figure 4. Both have phenyl groups, imide rings, and aromatic ether linkages. The Ultem has more aromatic ether structure than does the Kapton and, in addition, has gem-dimethyl groups (the two methyl groups attached to the carbon located between the phenyl rings). These additional structures, plus the existence of meta- as well as para-substitution on the benzene rings, all contribute to the thermoplastic properties of Ultem.

The IR spectrum for the nonirradiated Ultem is shown in figure 5(a), and the changes that occurred in the IR spectra with exposure to radiation are shown in figure 5(b). Each band's increase or decrease is an indication of the change in the relative amount of the molecular bond responsible for that band, but direct quantitative correlations should not be made for these IR spectra. The changes are identified in table I, where the column of band numbers corresponds to the set of numbers in figure 5(a).

Table I. Changes in Infrared Absorption Bands of Irradiated Film

Band number	Infrared band	Change
1	C-H (phenyl)	Increase
2	C-H (methyl)	Decrease
3	C = O	No change (1.6×10^9 and 4.0×10^9 rad) Decrease (6.0×10^9 rad)
4	Ether (aromatic)	Decrease
5	C-N	No change
6	Phenyl (tri- and di-substituted)	Decrease
7	Phenyl (Mono-substituted)	Increase
8	Imide ring	No change (1.6×10^9 and 4.0×10^9 rad) Decrease (6.0×10^9 rad)

Whereas the IR technique identifies the bonds that are broken or formed, the EPR technique identifies the corresponding radicals that are formed or combined for those particular bond changes. The EPR spectrum for the Ultem for the highest dose studied is presented in figure 6. The EPR spectrum of the nonirradiated Ultem is shown in the same figure. The spectrometer receiver gain required for the nonirradiated Ultem is 134 times that needed for the irradiated material. There are four radicals that are apparent in the irradiated Ultem EPR spectra. These radicals are indicated in figure 6, and their chemical structures are given in figure 7. Also shown in figure 7 are the structures of the phenyl radical and the atomic hydrogen radical. During the formation of the phenoxy radicals and the ketone radicals, phenyl radicals must have simultaneously been formed. The existence of the gem-dimethyl and cyclohexadienyl radicals necessitates that free hydrogen atoms be present in the material during the irradiation. Since the phenyl and hydrogen radicals are not apparent in the EPR spectra of the irradiated Ultem, these radicals must have decayed out prior to the recording of the EPR spectra.

The model of the radiation damage in the Ultem prior to the crosslinking is presented in figure 8. The

aromatic ether linkages (that is, the bonds shown in figure 4(a) between the oxygen and the phenyl groups on either side) were broken. These broken bonds resulted in the formation of phenoxy (para- and tri-substituted) radicals as shown in the top line of figure 8. The lineshape of the EPR signal from the phenoxy radicals had both Lorentzian and Gaussian characters; thereby, the existence of both para- (top left of fig. 8) and tri-substitution (top right of fig. 8) for the phenoxy radicals in the Ultem was verified. The bonds shown in figure 4(a) between the ketonic carbons (carbons with a double bond to an oxygen) and the phenyl/imide carbons (carbons that are both members of the six-carbon phenyl ring and the five-member imide ring) were also broken. Thus, as shown in the bottom line of figure 8, ketone radicals were formed.

At the same time that the phenoxy and the ketone radicals were formed, hydrogen atoms were stripped from the gem-dimethyl groups (the two methyl groups attached to the same main-chain carbon in fig. 4(a)) to form gem-dimethyl radicals. Some of these hydrogen atoms bonded, as shown in the top line of figure 8, with the unpaired electrons at the various phenyl radical sites that were formed when the ether linkages and the imide rings were broken. Other hydrogen atoms were trapped temporarily at a benzene ring; this trapping formed the cyclohexadienyl radicals as shown in the middle line of figure 8. This left behind four radicals—the para-substituted phenoxy, the tri-substituted phenoxy, the gem-dimethyl, and the ketone radicals—the decay of whose total density in vacuum is given in figure 9. Since the phenyl and hydrogen radicals had already combined, the remaining path for the radical decay observed in figure 9 was through radical-radical combinations of these four radicals. These combinations produced the crosslinkings that were responsible for the observed changes in the tensile properties of the Ultem.

There are $10 [n(n + 1)/2]$, where $n = 4$ possible unique molecular structures resulting from the pairwise combinations of the four radicals in the irradiated Ultem. These ten crosslinkages are shown in figure 10. Two gem-dimethyl radicals can join to form an ethylene group as shown in figure 10(a). A gem-dimethyl radical can join with a ketone radical to form an amide group (fig. 10(b)) or join with either of the two phenoxy radicals to form ether groups (fig. 10(c)). The phenoxy radicals can join ketone radicals to form urethane groups (fig. 10(d)) or join each other to form peroxide groups (fig. 10(e)). Finally, it is also possible for a ketone radical to join its own kind as shown in figure 10(f); however, this structure may not be very stable.

The IR spectrum for the nonirradiated Kapton is given in figure 11(a), and the changes in the bands due to the radiation are presented in figure 11(b). Compared with the Ultem IR data in figure 5(b), the changes in the Kapton (fig. 11(b)) are smaller and fewer. The two bands at 1780 and 1720 cm^{-1} for the symmetric and antisymmetric vibration modes of the carbonyl pair and the bands at 1270 and 1235 cm^{-1} for the aromatic ether bonds all decreased by very small amounts. No other changes occurred.

The EPR spectrum for the irradiated Kapton for the 6.0-gigarad dose is given in figure 12. Also in figure 12 is the EPR spectrum for the nonirradiated Kapton. The gain used for the spectrum of the nonirradiated material is 25 times that used for the spectrum of the irradiated material. There are three radicals present compared with the four present in the irradiated Ultem. Two of the radicals are the same as those found in the Ultem: the phenoxy radical and the ketone radical. The third radical, however, is different from those that appeared in the EPR signal for the irradiated Ultem. It is identified to be the phenyl radical (refs. 6 through 17). The chemical structures of these three radicals are shown in figure 13.

The decay of the total density of these three radicals for the irradiated Kapton is presented in figure 14. Again, as for the Ultem, the decay is due to radical-radical combination. However, as is explained in the following discussion, the radical-radical combination process is different in the Kapton, and it is this difference that explains the lack of an effect of the radiation on the tensile properties of Kapton.

It is worthwhile noting that an integration of the area under the entire EPR absorption curve leads to the determination of the total radical density. The radical densities, before and after the radiation exposures, indicated that there was greater change for the Ultem than for the Kapton. Plots of the total radical density, as a function of the total electron radiation dose, for the two polyimides are given in figures 15 and 16, respectively. The Ultem data showed over 2 orders of magnitude increase, whereas the Kapton data showed less than 1 order of magnitude increase. This comparison, like the comparison of the IR data in figures 5(b) and 11(b), suggests that a larger measurable radiation effect took place for the Ultem than for the Kapton.

A model of the initial radiation damage mechanisms that took place in the Kapton is presented in figure 17. A phenyl radical (top left of fig. 17) and a phenoxy radical (top right of fig. 17) were formed when the ether linkage bond was broken. Another phenyl radical and a ketone radical were formed when

the bond between the phenyl carbon and the ketonic carbon in the imide ring was broken, as shown at the bottom of figure 17(c). Unlike the case of the Ultem, there was no aliphatic site for dehydrogenation; therefore, there was no hydrogen readily available for combining with the phenyl radicals. Consequently, in both instances, the phenyl radical was free to recombine with its partner radical. In effect, there was little net change in the structure of the bonding because self-mending occurred. Consequently, there was little buildup, at any time, of broken bonds and radicals; therefore, there was just a small change in the magnitude of the absorption bands for those IR spectral changes that did occur and there was less than an order of magnitude increase in the total EPR radical density. It followed that since there was little net change in the molecular structure, no changes in the tensile properties of the irradiated Kapton were observed.

Although the model and the explanation for what occurred in the irradiated Kapton are not complicated, their formulation depended on the insight that had been gained by the previous analysis of the data for the Ultem film specimens that had been exposed to the same radiation conditions. The combination of the decrease in the aliphatic C-H IR band at 3060 cm^{-1} , the absence of the phenyl radicals, and the presence of the gem-dimethyl radicals for the Ultem—in contrast to the presence of the phenyl radicals for the Kapton—provided the key finding: In the case of the Kapton, self-mending of the radiation damage was occurring. Had there been other mechanisms taking place, such as both permanent chain scissioning and crosslinking in essentially equal quantities, then additional IR and EPR spectral changes would have been observed.

The explanation for the effects of radiation on a complex molecular structure, such as Kapton, via an understanding of the radiation effects on an equally complex molecular structure, such as Ultem, is not traditional. The traditional approach is to use much simpler structures for the modeling of complex systems. Although the traditional approach should not be abandoned, this study has pointed out, in the case of studying radiation effects, that the simpler structure is not necessarily the only key for providing an understanding. Indeed, it is possible that the lack of similar complexity in the simpler structures may render them of little value in understanding the radiation durability of the more complex polymeric systems.

Additional observations can be made from the data presented herein for the two irradiated polyimides. Aromatic structures are frequently regarded to be more durable to radiation than are aliphatic

structures. Although this study does not dispute this, it does indicate that bonds to aromatic structures can be readily broken and that the extent of the durability depends on the other functionalities present. This is in contrast to the concept that the introduction of aromatic structure by itself enhances durability. Finally, it is the absence of aliphatic hydrogen, rather than the presence of aromatic structure, that accounts for the enhanced radiation durability of the Kapton as compared with the Ultem.

Concluding Remarks

Two polyimide materials, Kapton and Ultem, in the form of films, have been compared for the durabilities of their tensile properties to electron radiation doses expected for 30-year missions in geosynchronous orbit. The infrared and electron paramagnetic resonance spectra before and after exposure have also been studied for the effects of the radiation doses on the molecular structure of the two materials.

The spectroscopic data showed that the primary electron-radiation-generated change in the tensile properties of the Ultem, a large reduction in the total elongation, was due to crosslinking that followed the capture by phenyl radicals of hydrogen atoms removed from gem-dimethyl groups. In contrast, the tensile properties of Kapton were not affected by radiation because radical-radical recombination, a self-mending process, took place.

The explanation for the effects of the radiation on the Kapton was, in part, based on the model for the effects of the radiation on the Ultem. This study has pointed out, at least for the case of studying radiation effects, that studying a simpler model structure is not necessarily the only key for providing understanding.

The development of new, radiation-durable polymers can benefit from the findings of this study by producing polymeric systems that do not have moieties, such as methyl groups that are a source of hydrogen, that can inhibit self-mending.

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Hampton, VA 23665-5225
December 10, 1986

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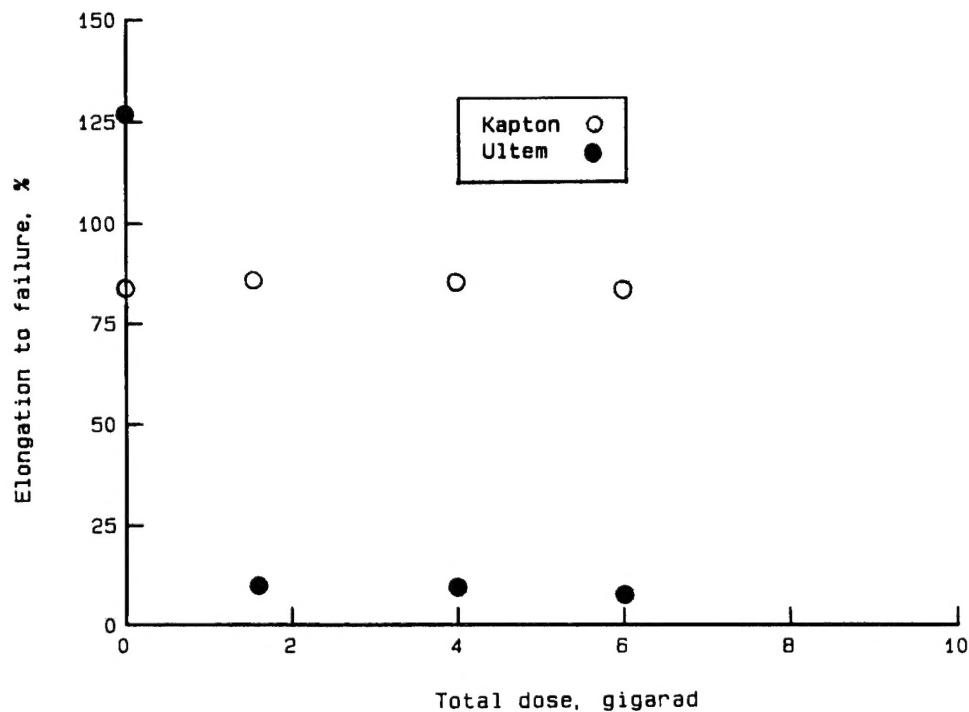


Figure 1. Percent total elongation to failure as a function of total dose for Ultem and Kapton films.

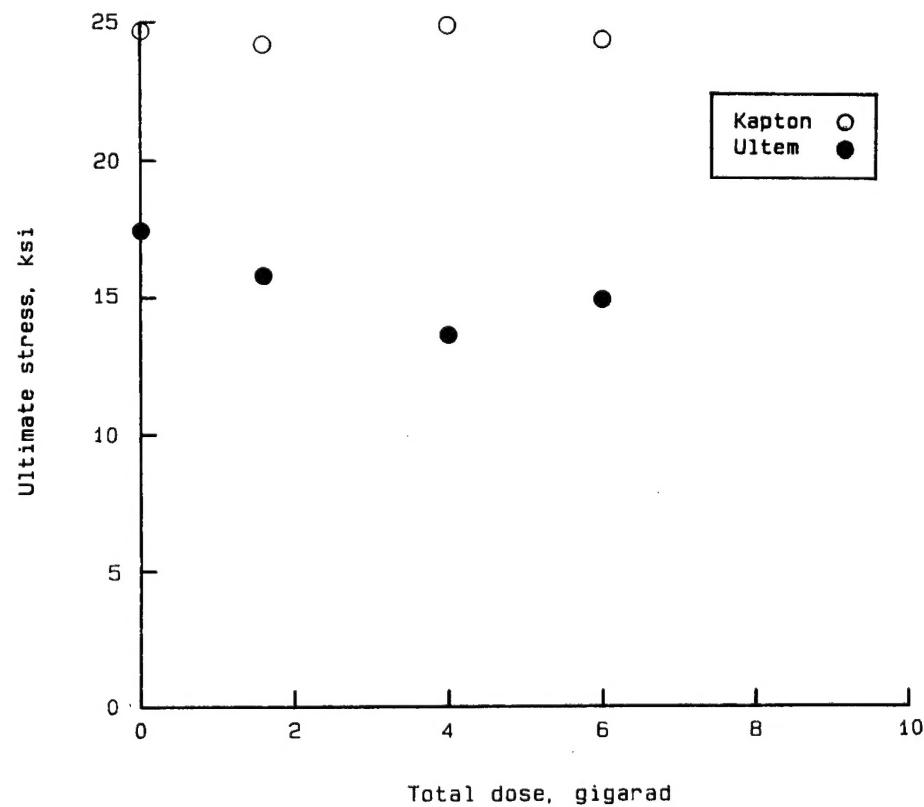


Figure 2. Ultimate stress as a function of total dose for Ultem and Kapton films.

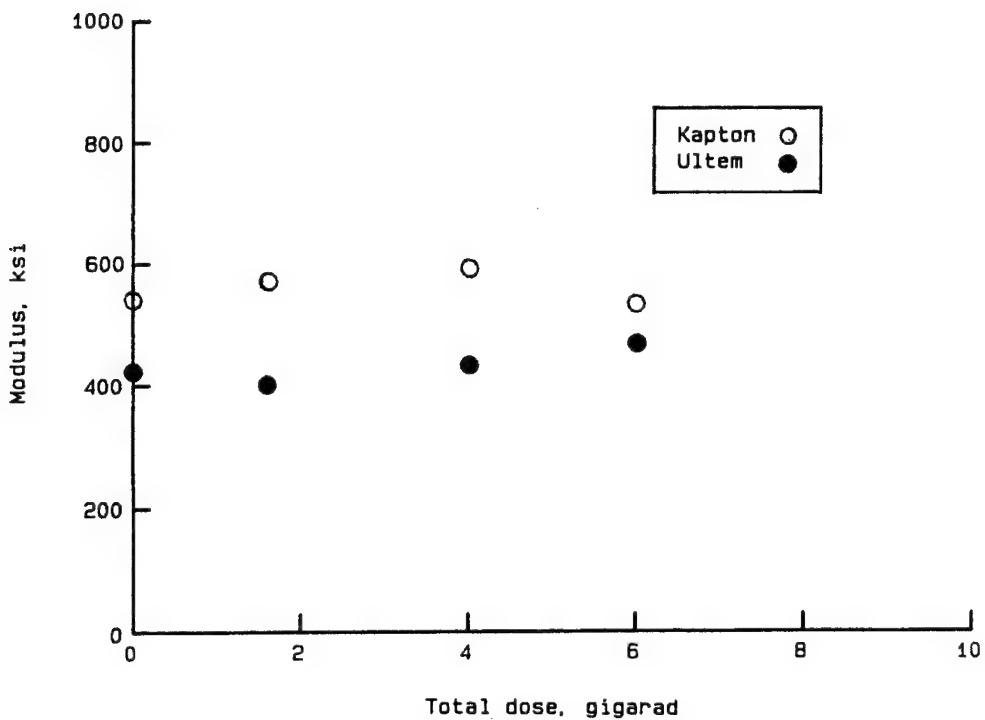
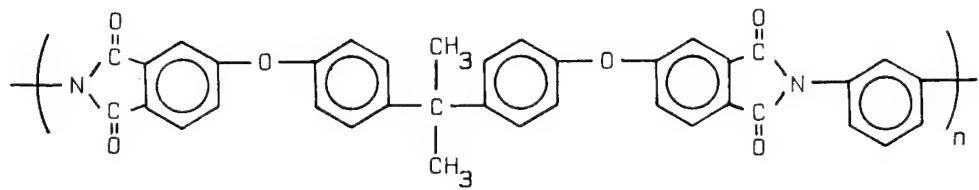
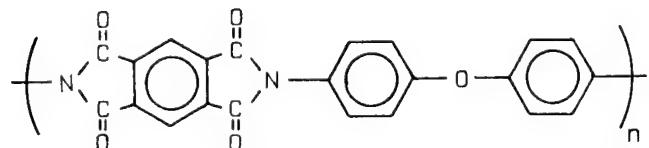


Figure 3. Modulus as a function of total dose for Ultem and Kapton films.

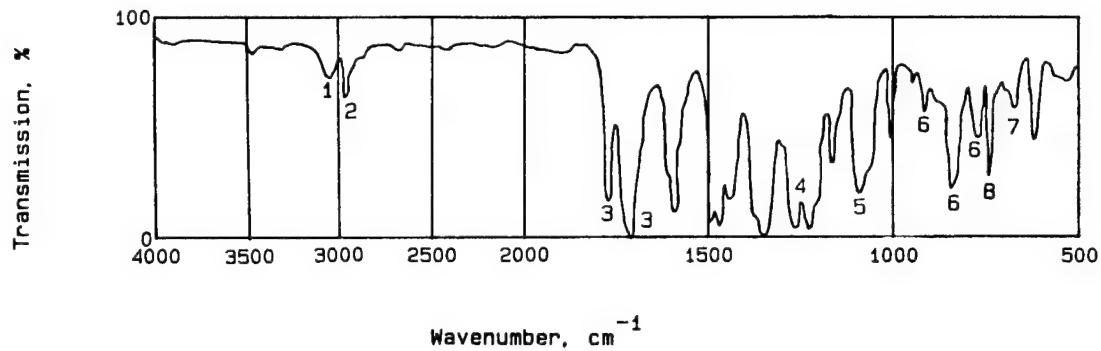


(a) Ultem.

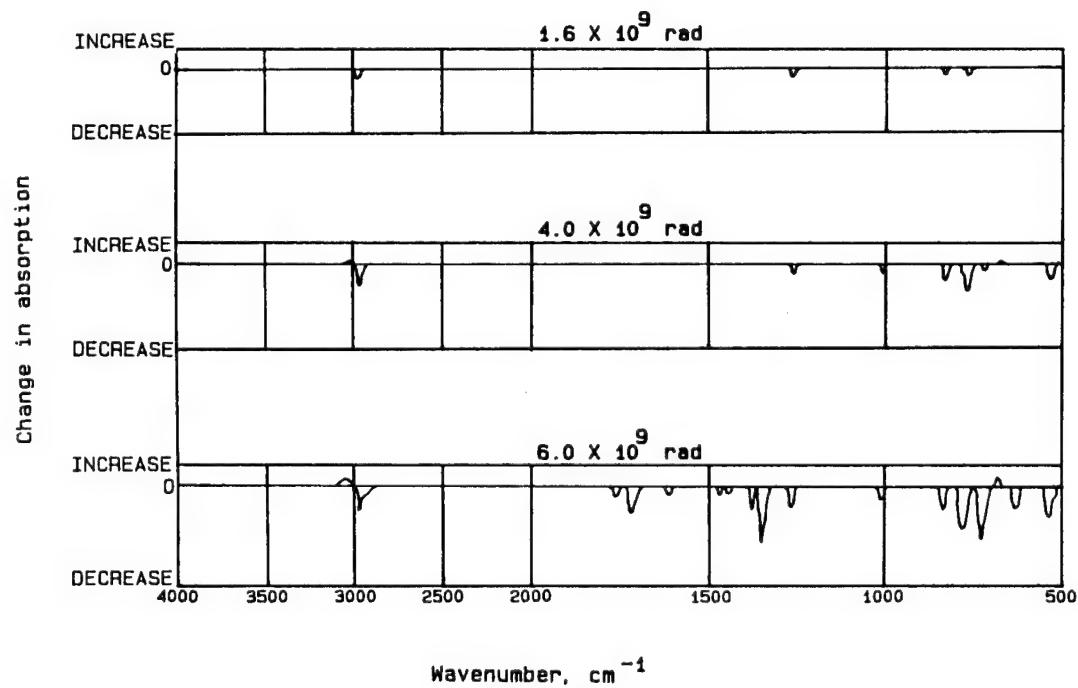


(b) Kapton.

Figure 4. Molecular structures of nonirradiated Ultem and Kapton.



(a) Infrared spectrum.



(b) Difference spectra.

Figure 5. Nonirradiated infrared spectrum and radiation-generated difference spectra for Ultem. Numbered bands are identified in table I.

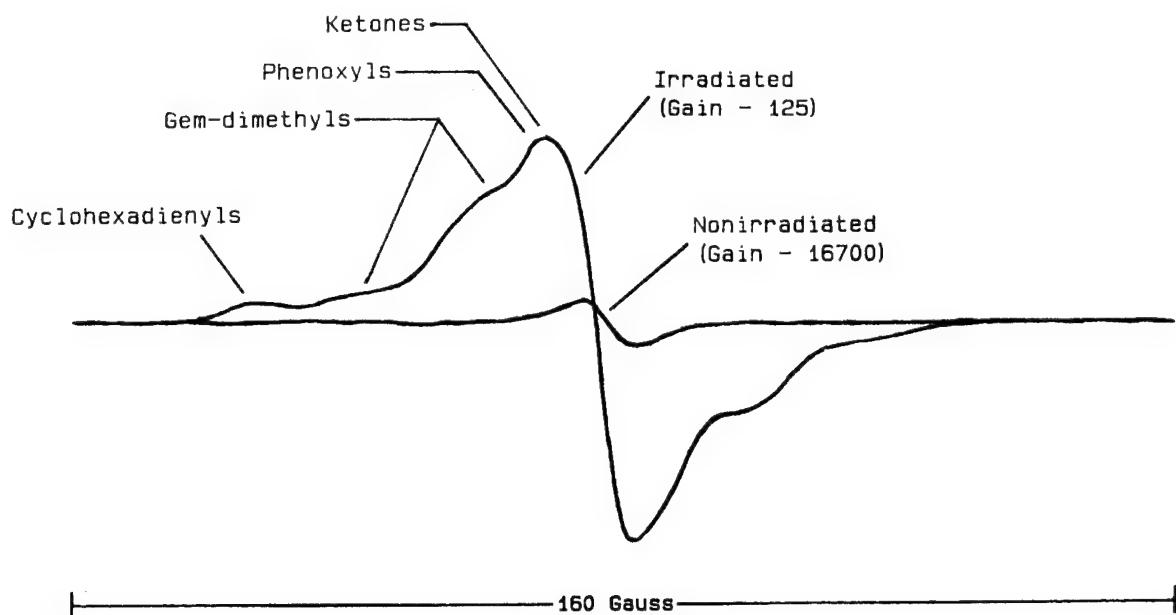


Figure 6. Identification of radicals in irradiated Ultem EPR spectrum, under vacuum, and spectrum before irradiation.

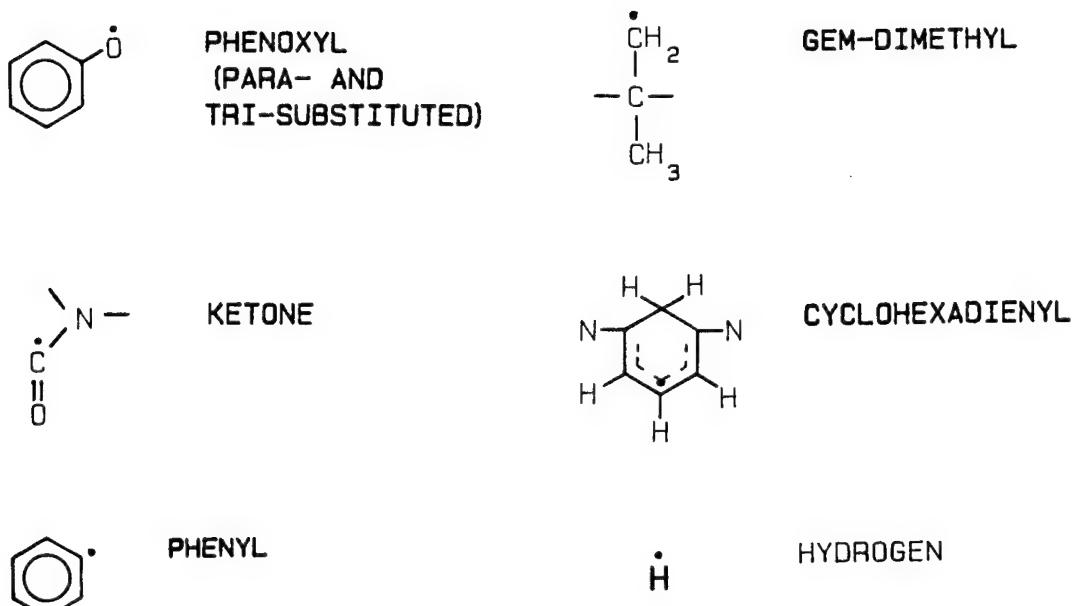


Figure 7. Structures of radicals in irradiated Ultem under vacuum.

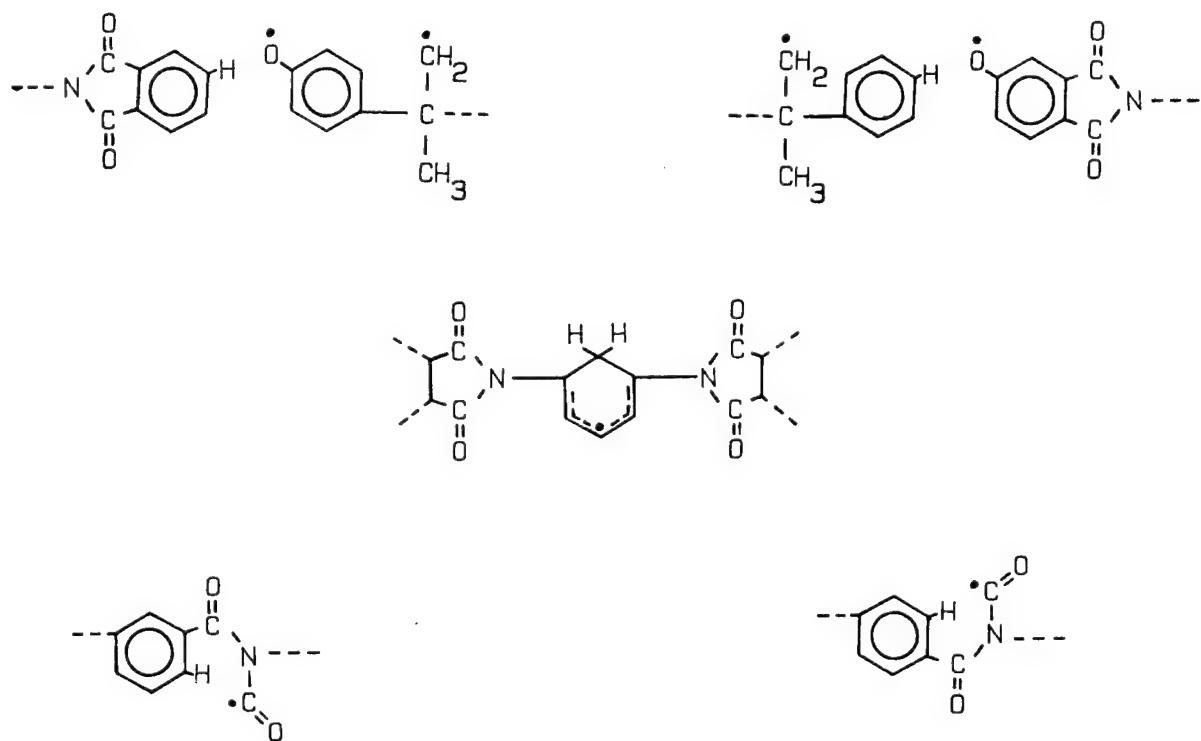


Figure 8. Model of radiation damage in Ultem film prior to crosslinking.

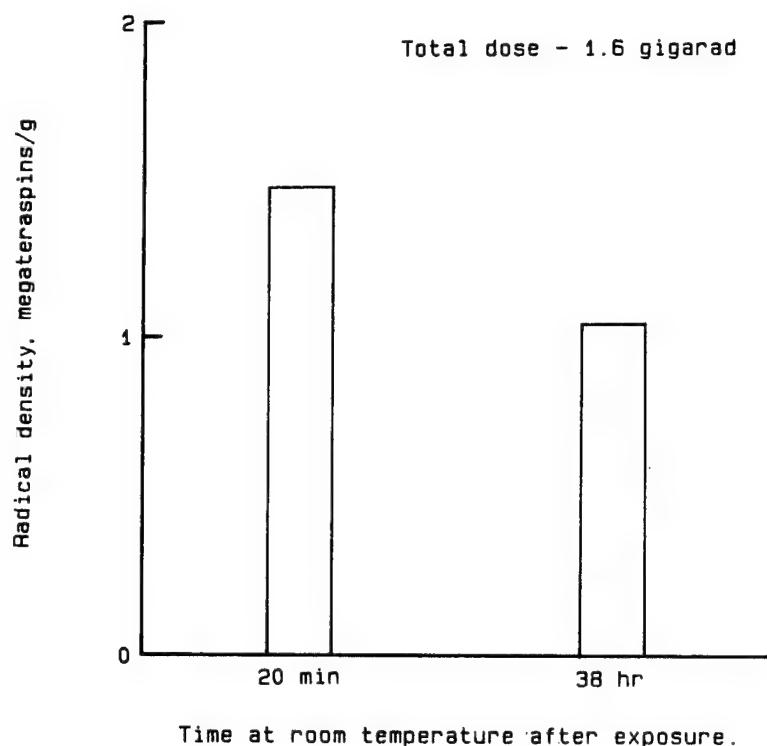


Figure 9. Total radical decay in vacuum for irradiated Ultem.

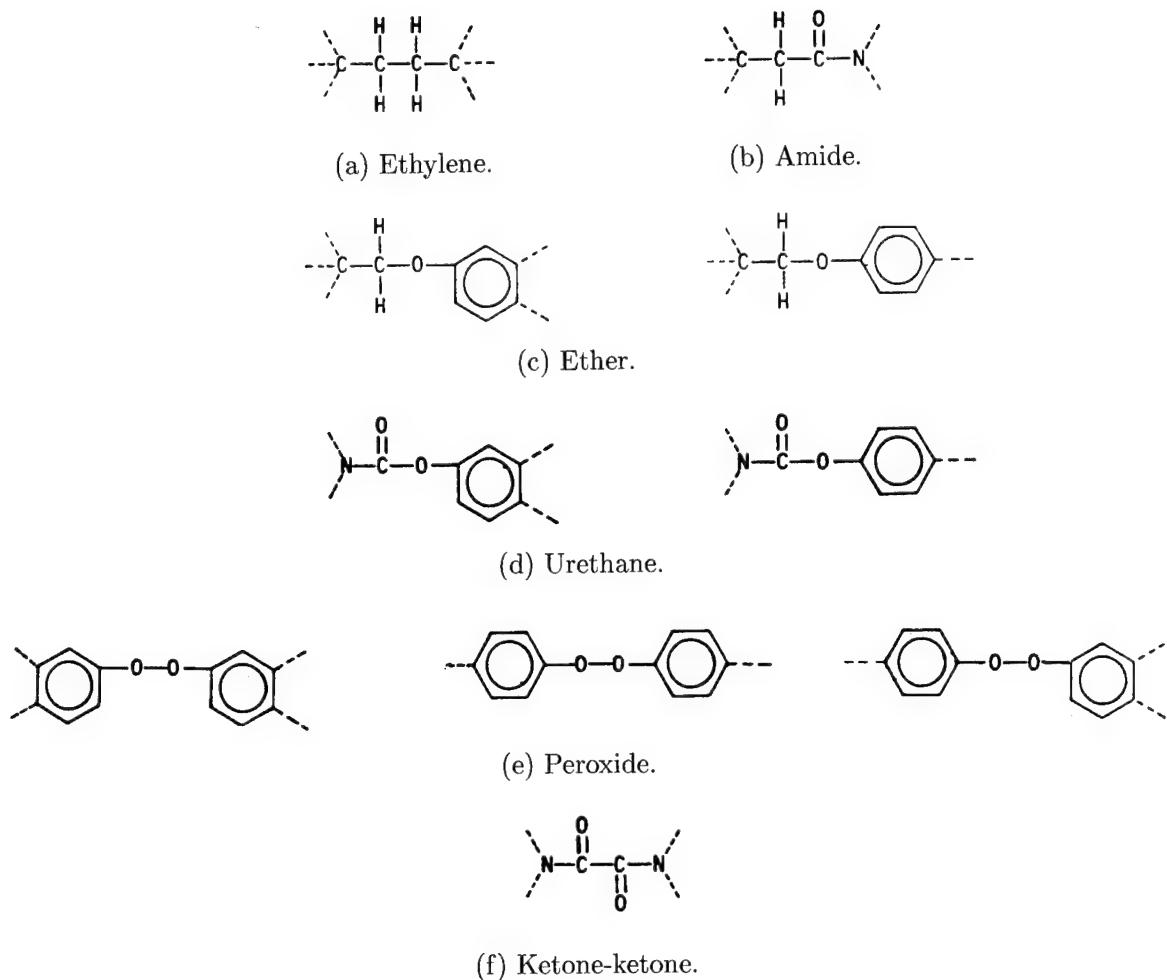
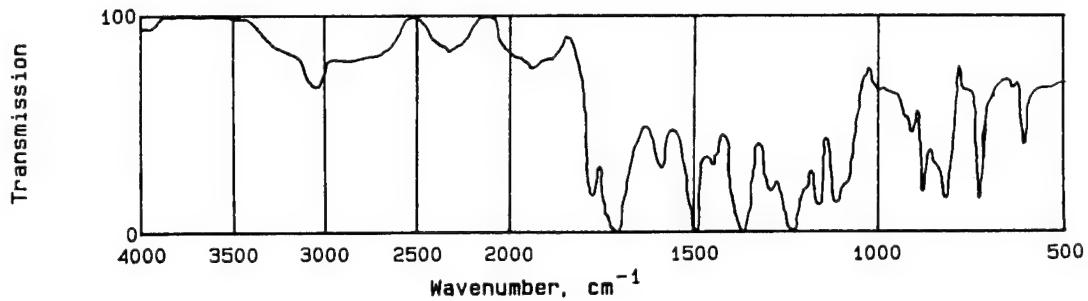
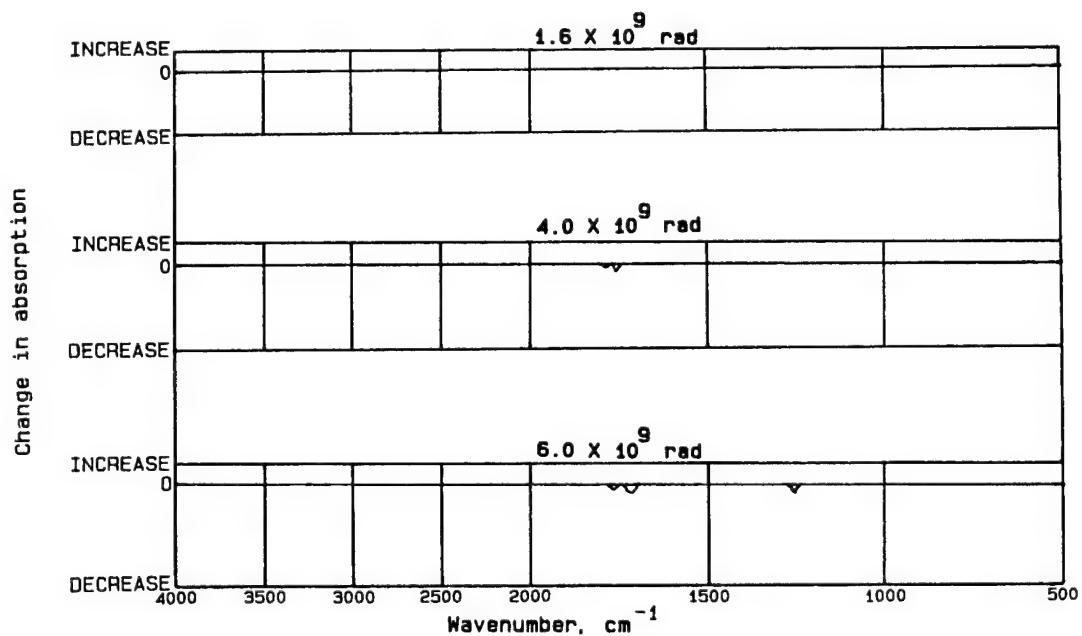


Figure 10. Possible molecular structures in irradiated Ultem due to the crosslinking of the radiation-generated radicals.



(a) Infrared spectrum.



(b) Difference spectra.

Figure 11. Nonirradiated infrared spectrum and radiation-generated difference spectra for Kapton.

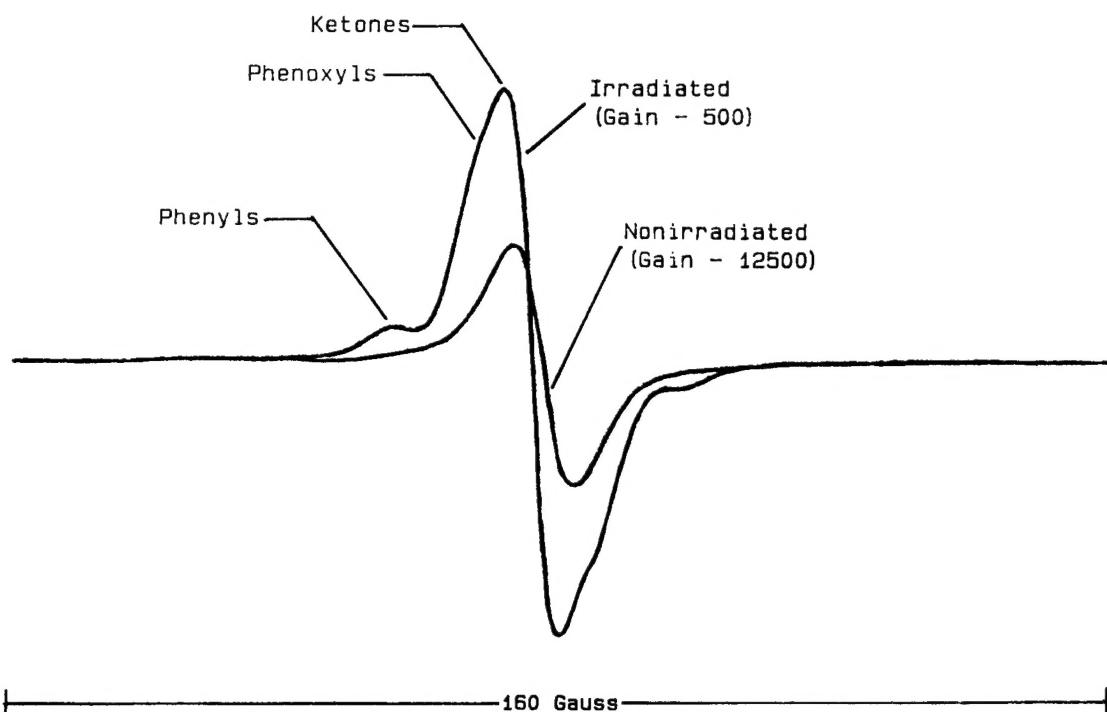


Figure 12. Identification of radicals in irradiated Kapton EPR spectrum, under vacuum, and spectrum before irradiation.

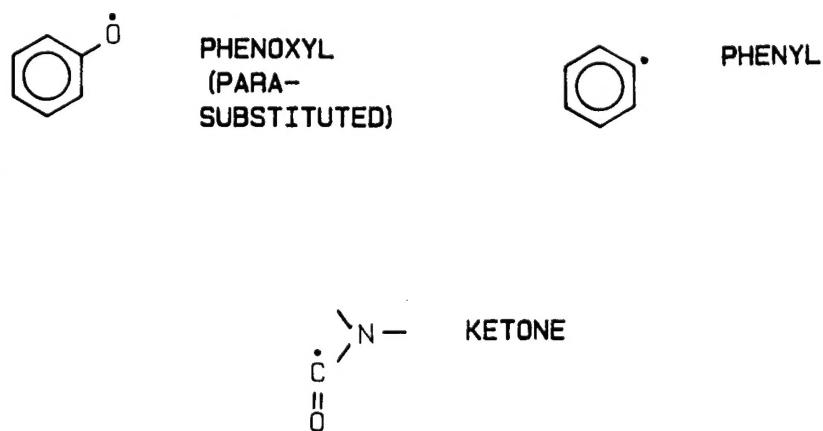


Figure 13. Structures of radicals in irradiated Kapton under vacuum.

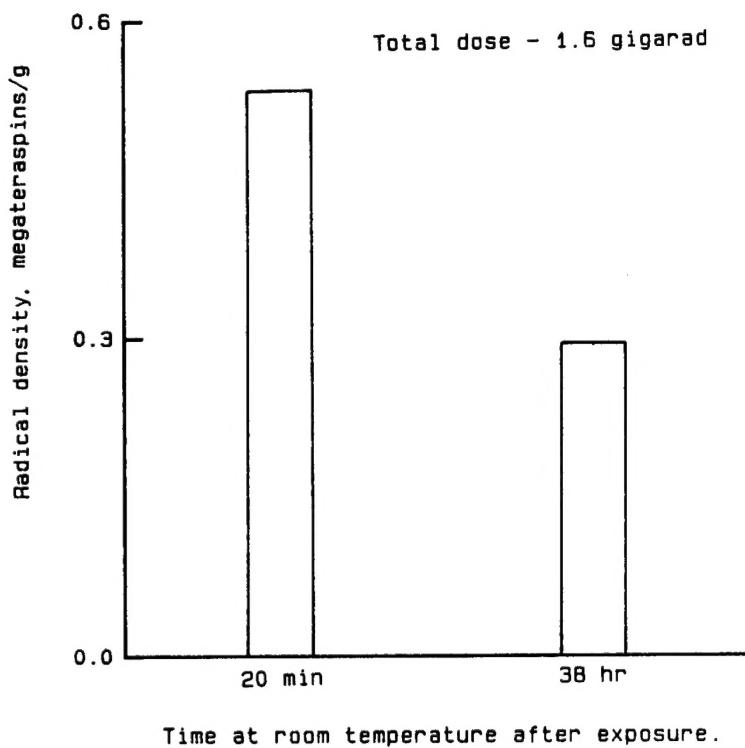


Figure 14. Total radical decay in vacuum for irradiated Kapton.

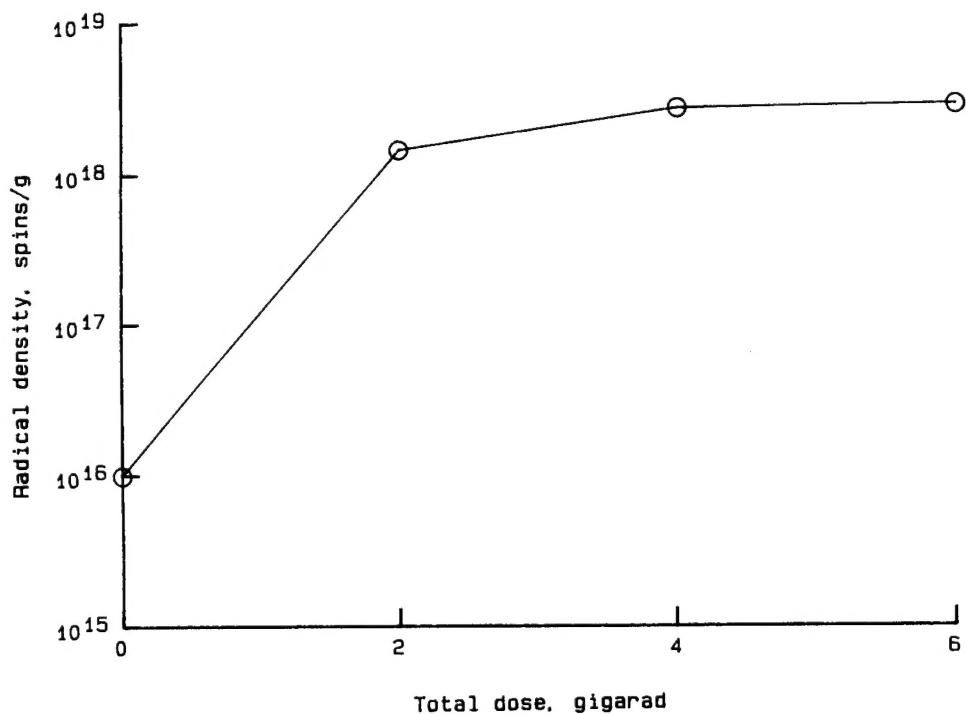


Figure 15. Total radical density as a function of total electron radiation dose for Ultem film.

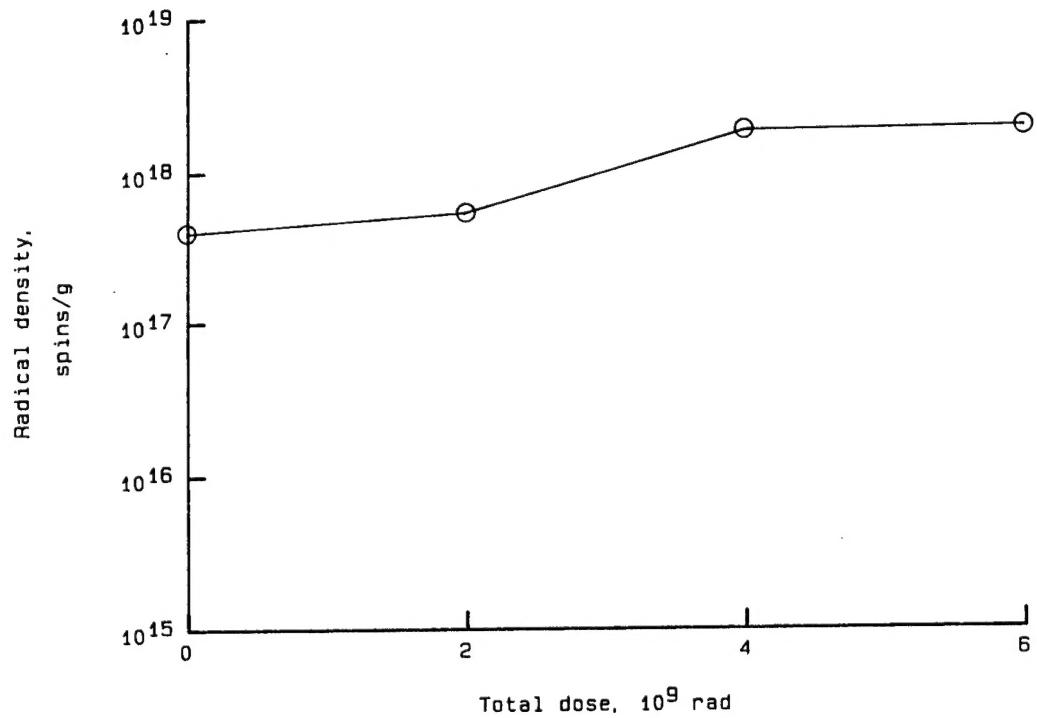


Figure 16. Total radical density as a function of total electron radiation dose for Kapton film.

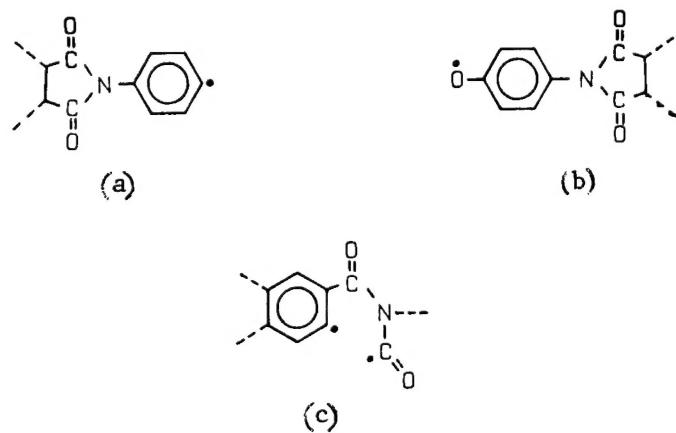


Figure 17. Model of radiation damage in Kapton film prior to self-mending.

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16. Abstract A comparison of the radiation durabilities of the mechanical properties of a polyetherimide and a polyimide is made. A model for each polymer, developed from analyses of infrared and electron paramagnetic resonance spectroscopic data, is provided to explain the basic mechanisms taking place upon exposure to electron radiation. The molecular model for the polyimide was, in part, established from the modeling for the polyetherimide. In this respect, the report demonstrates techniques for understanding the durability of one complex polymer based on the understanding of a different and equally complex polymer.				
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